INTERLABORATORY
STUDY 91-5

DISSOLVED ORGANIC CARBON
AND
TOTAL ORGANIC CARBON
IN
REAGENT WATER AND EFFLUENT

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DISSOLVED ORGANIC CARBON AND TOTAL ORGANIC CARBON IN REAGENT WATER AND EFFLUENT

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1 SUMMARY OF INTERLABORATORY STUDY 91-5

Interlaboratory Study 91-5 was initiated as part of an on-going program of laboratory performance management studies conducted by the Quality Management Office of Laboratory Services Branch, Environment Ontario. It assesses the performance of participating laboratories for the analysis of Dissolved Organic Carbon (DOC) and Total Organic Carbon (TOC) in spiked reagent water and in effluents. Twenty-nine laboratories (government, industry, and commercial) agreed to participate in this study. Participants received two spiked and one unspiked sample in reagent water and in a Sewage Treatment Plant effluent. Results were received from twenty-four participants for DOC and from twenty-five participants for TOC. The test groups are MISA Analytical Test Group 5a (DOC) and 5b (TOC)[1].

Most participants were able to achieve acceptable performance on both the reagent water and the effluent samples for both DOC and TOC analysis. Most participants that were flagged had erratic performance, i.e. one result agreed with the target and interlaboratory mean and median while the other result differed from the target and interlaboratory mean and median. Table I below summarizes the grouping of the participating laboratories.

TABLE I
SUMMARY OF STUDY FINDINGS - DOC AND TOC

Rank	DOC n=24				TOC		
Matrix	**Reagent Water**		**Effluent**		**Reagent Water n = 24**		**Effluent n = 25**
Evaluation Point	**Target**	**Median**	**Target**	**Median**	**Target**	**Median**	**Target/ Median**
Acceptable	14 (58%)	13 (54%)	19 (79%)	16 (67%)	19 (79.5%)	18 (75%)	21 (84%)
Out of Control or Erratic	6 (25%)	5 (21%)	2 (8.5%)	4 (17%)	2 (8%)	2 (8%)	2 (8%)
Low (Slope or Intercept)	3 (13%)	3 (12.5%)	2 (8.5%)	2 (8%)		1 (4%)	1 (4%)
High (Slope or Intercept)	1 (4%)	3 (12.5%)	1 (4%)	2 (8%)	3 (12.5%)	3 (13%)	1 (4%)

2 INTRODUCTION

Interlaboratory performance studies are conducted to assess the comparability and accuracy of data among different laboratories. These studies are useful for the identification of biases, precision and accuracy problems. Participation in such studies can serve as a guide for improving individual laboratory performance and maintaining performance standards. The Quality Management Office, Laboratory Services Branch, Environment Ontario has instituted an on-going program of interlaboratory studies to assess and enhance the performance of environmental laboratories providing analytical services.

This study was designed to assess the analytical variability among laboratories for the analysis of Dissolved Organic Carbon (DOC) and Total Organic Carbon (TOC) in spiked reagent water and effluent. These tests were chosen from the MISA (Municipal and Industrial Strategy for Abatement) General Regulation[1]. Participants were requested to use methods which conformed to the MISA analytical principles and protocols given in the General Regulation[1].

Twenty-nine laboratories (government, industry, and commercial) agreed to participate in this study. Results were received from twenty-four participants for DOC and from twenty-five participants for TOC. A list of participants is given in Appendix 2. Each participant was assigned a unique identification code to maintain confidentiality.

Section 3 describes sample preparation, sample distribution, analytical methodology, and data evaluation procedures. Final results are tabled in Appendix 1 and discussed in Section 4.

3 PROCEDURE

3.1 Preparation of Samples

A spiking solution of Potassium Hydrogen Phthalate was prepared in distilled, deionized water and sealed into ampoules.

The reagent water blank was prepared by measuring 20 litres of distilled, deionized water (DDW) into a stainless steel tank. 12 mL of Sulfuric Acid was added to the tank and stirred for 10 - 20 minutes. The pH was verified as <2.0 (actual result = 1.9). The solution was dispensed into 500 mL PET500 bottles and labelled "D7".

In a similar manner as above, the reagent water Low Spike was prepared by including an appropriate amount of the Spiking Stock (using volumetric glassware) in the 20 litre DDW solution. After stirring, the solution was dispensed into 500 mL bottle and labelled "D8". The reagent water High Spike was made in the same manner and labelled "D9".

A bulk sample of effluent was collected from the Lakeview Sewage Treatment Plant on October 31, 1991. The effluent was filtered prior to use in preparing the interlaboratory study samples. The unspiked effluent and the two spiked effluent samples were prepared in the same manner as the reagent water samples above. The unspiked effluent was labelled "E7", the Low Spike was labelled "E8" and the High Spike was labelled "E9".

Spiking levels for all samples are included in Tables 1 and 2 in Appendix 1.

3.2 Sample Distribution

Samples were packed and stored overnight at 4°C. Samples were shipped the following day by Purolator Courier to the participating laboratories. A list of the laboratories receiving sample sets is given in Appendix 2. Samples were shipped on November 5, 1991. A copy of all correspondence is also included in Appendix 2.

3.3 Analytical Methodology

Participating laboratories were requested to analyze the samples using routine in-house methods that complied with the principles and protocols outlined in Schedule 3 of the MISA General Regulation[1]. Participants were requested on the report form provided (Appendix 2) to summarize their Sample Preparation Principles and Instrumental Measurement Method Principles. All participants were assigned a unique identification code.

3.4 Data Evaluation Techniques

Results were submitted to the Quality Management Office, LSB in written form. All data were manually entered by laboratory code into an electronic spreadsheet. Some participants were able to analyze for only DOC or TOC. Individual reasons provided by each participant are included in the Individual Laboratory Performance described in Section 4.

The participating laboratories who reported results were mailed a copy of the tables of results on January 15, 1992.

The mean, median and standard deviation were calculated for each parameter in each sample and are included in Tables 3 and 4, Appendix 1.

The spiked reagent water samples and the spiked effluent samples were evaluated using the technique outlined in References 2 and 3. This results in identifying an area of acceptable performance based on the precision of the participating laboratories. The DOC and TOC results were evaluated separately. The results for the spiked reagent water samples (D8 and D9) were evaluated as reported and re-evaluated after subtracting any reported values for the unspiked reagent water sample (D7). The background level of DOC or TOC reported in the unspiked effluent sample (E7) was subtracted from each participants' results for the spiked samples (E8 and E9) prior to using the King-Selliah Technique[2,3]. All data sets were evaluated versus the target and versus the interlaboratory median. This Automated Evaluation Technique[3] is summarized as follows:

1. Two samples at different concentrations are split among a number of laboratories and analyzed as requested. Results are entered into an electronic spreadsheet and programs (macros) are coded to perform the manipulations. The evaluation technique is performed after the participants have verified their results (see above).

2. High sample data evaluation:
    - i) Reject all results which differ from the median (H_m) by more than 10%.
    - ii) Calculate median (H), mean and standard deviation (S_h).
    - iii) Reinclude results if within 3 times S_h.
    - iv) Reiterate ii) and iii) until no further results are included.
    - v) Calculate relative standard deviation of final selected results (CV_h).

3. Low sample data evaluation:
    - i) Use 3 x CV_h x median(L_m) to exclude possible outliers.
    - ii) Calculate median (L), mean and standard deviation (S_l).
    - iii) Reinclude results if within 3 times S_l.
    - iv) Reiterate ii) and iii) until no further results are included.

4. Paired sample performance criteria:
    - i) Examine ratio of S_h/S_l: If <2, use results as reported in concentration units. Otherwise convert results to percent recovery based on target value, if known, or use recovery relative to median value - H, L.
    - ii) Prepare paired sample scatter diagrams of all results (Youden plots[4]).
    - iii) Calculate perpendicular distances from each point to the two 45° lines (Slope and Intercept error lines) and select the lesser of the two perpendicular distances (PD).
    - iv) Determine the median PD.
    - v) Determine the mean of all PD values less than 2.5 times the median and use this mean to estimate the repeatability S_w.
    - vi) Set warning limits for repeatability: 2 times S_w.
    - vii) Set control limits for repeatability: 3 times S_w.
    - viii) Set warning limits for possible bias: 3 times S_w (same as vii).
    - ix) Set control limits for possible bias: 4.5 times S_w.

5. Code performance and summarize in table
    - i) In upper left or lower right quadrant - Erratic
    - ii) In lower left or upper right quadrant - Biased Low or High
    - iii) On horizontal or vertical axis - Out of Control
    - iv) On diagonal line through origin - Slope or Standard problem
    - v) On diagonal line not through origin - Intercept or blank problems.

The performance evaluation of each participant using the above technique is summarized in Tables 5 and 6. The graphical results are presented in Figures 1-8, Appendix 1.

The results for the spiked effluent samples (E8 and E9) were also plotted using the Youden technique[4] without subtracting the results for the unspiked effluent (E7). These are included in Appendix 1 as Figures 9 and 10.

4 DISCUSSION

OVERVIEW OF INTERLABORATORY PERFORMANCE

Most participants did not detect any DOC in the unspiked reagent water sample (D7). However, six laboratories did report a value of DOC in sample D7. The King-Selliah Technique[3,4] was applied to the data set using both the uncorrected and the corrected results for five of the laboratories reporting a value for D7. The correction was not applied to Laboratory 9040's results as it would have resulted in negative results (See Individual Performance below).

The interlaboratory mean and median (both corrected and uncorrected) for the Low Spike in reagent water (D8) show good agreement with the target. The interlaboratory mean and median for the High Spike in reagent water (D9) are slightly lower than the target value. When the data sets were evaluated[4] versus the median as opposed to the target, there was a slight shift in the centre point of the area of acceptable performance. The evaluation versus the median resulted in a smaller value for S_w (Table 3), so that two participants that were Acceptable versus the target were shifted as Warning High and one participant that was Erratic versus the target moved to Warning: Slight Imprecision (Table 5). See Individual Performance below for discussion of possible causes for these changes in rank.

Subtracting the reported value for D7 from the spiked samples D8 and D9 did not affect the laboratory's ranking in some instances. For one laboratory performance shifted outside the acceptable area while for others performance improved. Possible reasons for these affects are discussed individually below. These variations in effect caused by correcting for the results for the unspiked sample indicate that caution is required when applying a correction factor. Data must be examined carefully so that the use of a correction factor is justified.

To evaluate the DOC results in the spiked effluent samples (E8 and E9), the reported values for the unspiked effluent sample (E7) were subtracted from the E8 and E9 results. This was necessary as the background level of DOC in the effluent is considerably higher than the spike level in both spiked samples.

As with the reagent water sample, the interlaboratory mean and median (background corrected) of the Low Spike in effluent (E8) showed good agreement with the target value. The variability (standard deviation) was very similar for both matrices at this level (Table 3). The mean and median (background corrected) of the High Spike in effluent (E9) were lower than the target value. Variability was greater in the effluent matrix than the reagent water for the higher spiked samples (Table 3).

The King-Selliah Technique[4] was applied only to the corrected effluent sample results. When evaluated versus the median as opposed to the target, there is a noticeable shift in the centre point for the area of acceptable performance (Figures 2 and 4). This is due to the lower value for the interlaboratory median for E9 as noted above. As a result, several laboratories who were Acceptable when evaluated versus the target being flagged as Out of Control or High when evaluated versus the median (Table 5). Laboratories that had acceptable agreement with the target in these circumstances should not consider that their performance is inadequate, but should continue to maintain high standards of performance.

Similar patterns are noted for the TOC results in reagent water as for the DOC results noted above. For the effluent samples, there was no difference in the ranking of the participants, whether they were evaluated versus the target or the median (Table 6).

The spiking level used for the effluent samples was considerably lower than the background level of DOC and TOC. The ranking assigned to the participants using the King-Selliah Technique[4] was done on the corrected results and is at a much lower level than the samples were actually analyzed. To determine the interlaboratory consensus, the results for the spiked effluent samples (E8 and E9) were plotted without subtracting the value for the unspiked sample (E7) using a Youden two-sample plot[4]. This was done for both DOC (Figure 9) and TOC (Figure 10), with each figure divided into quadrants based on the interlaboratory medians. Participants whose results appeared noticeably different from the main group were identified.

The results in Figures 9 and 10 show good within-laboratory precision for most laboratories for both DOC and TOC, as the majority of points lie on the diagonal line between the origin and the intersection of the medians. The data points are tightly clustered and the main source of difference among these laboratories can be attributed to a difference in standards.

INDIVIDUAL LABORATORY PERFORMANCE

Laboratory 9002

This laboratory analyzed only for TOC. They had Acceptable Performance for all samples in both matrices with no contamination in the unspiked reagent water sample (D7).

Laboratory 9003

This laboratory analyzed only for DOC. They had Acceptable Performance for all samples in both matrices with no contamination in the unspiked reagent water sample (D7).

Laboratory 9005

This laboratory had Acceptable performance for both DOC and TOC in all samples in both matrices with no contamination in the unspiked reagent water sample (D7).

Laboratory 9006

This laboratory had Acceptable performance for both DOC and TOC in all samples in both matrices with no contamination in the unspiked reagent water sample (D7).

Laboratory 9010

Laboratory had no contamination of the unspiked reagent water sample (D7). Their results for DOC were Out of Control or Erratic in both matrices (Table 5). Their result for D9 was low relative to the interlaboratory mean and median while their result for E9 was high relative to the interlaboratory mean and median. Results for D8 and E8 had good agreement with the interlaboratory mean and median. The same effect was noted for their results for TOC (Table 6). Sample E9 may have been contaminated, but no clear explanation for the low results for sample D9 are evident.

Laboratory 9011

Laboratory 9011 reported a result for the unspiked reagent water sample (D7) for DOC. When evaluated versus the target, they had Acceptable performance. When evaluated against the median, they are ranked Warning High (Table 5). When their result for D7 was subtracted from D8 and D9 and evaluated versus the median, their rank improves to Acceptable. This apparent improvement in performance versus the median is affected by the lower interlaboratory median value for D9 relative to the target value. As the flag was a warning, overall performance is acceptable. They had Acceptable performance in the effluent samples.

For TOC, Laboratory 9011 did not report a value for the unspiked reagent water sample (D7). They had Acceptable performance versus the target. When the reported values for D7 were not subtracted from samples D8 and D9, they had Acceptable performance versus the median. When the values for D7 were subtracted (for other participants), oeverall performance improved and the value of S_w became smaller (Table 4), resulting in a tighter area of acceptable performance. Laboratory 9011 becomes flagged as Warning High with an Intercept problem under the latter condition. As the flag was a warning, overall performance is acceptable. They had Acceptable performance in the effluent samples.

Laboratory 9012

This laboratory did not report any result for the unspiked reagent water sample (D7). They were flagged as Out of Control for DOC in the reagent water samples and Erratic in the Effluent samples (Table 5). For D8 and E9, they were high relative to the interlaboratory mean and median and the target. For sample D9 and E8, the had good agreement with the interlaboratory mean and median and the target. More consistent application of their method is recommended.

For TOC, they were flagged as Out of Control or High with Intercept problems in the reagent water samples (Table 6). For the effluent samples, they had Acceptable performance for TOC. However, their results for the effluent samples are higher than many of the other participants and show a high bias (Table 2, Figure 10). This suggests a bias in their calibration standards and should be checked using reference materials.

Laboratory 9015

This laboratory was unable to report any results for this study.

Laboratory 9016

This laboratory analyzed only for DOC. They had Acceptable Performance for all samples in both matrices with no contamination in the unspiked reagent water sample (D7).

Laboratory 9019

Laboratory 9016 was flagged as Warning Low with Slope or Intercept problems for both the reagent water and the effluent samples (Table 5). This suggests a bias in their calibration standards and should be checked using reference materials.

Laboratory 9016 had Acceptable performance for TOC in the reagent water samples (Table 6), but were flagged Low in the effluent samples. Their results for the effluent samples show little difference between the spiked and unspiked samples (Table 2), so that when the amount reported in E7 was subtracted from

E8 and E9, they were flagged low. This suggests that their method is not as sensitive for the matrix used in this interlaboratory study.

Laboratory 9020

Laboratory 9020 reported a value for the unspiked reagent water sample for DOC and TOC. They had Acceptable performance for both tests in the reagent water, and their rank did not change when their reported result for D7 was subtracted from D8 and D9 (Tables 5 and 6). The had Acceptable performance for DOC and TOC in the effluent samples.

Laboratory 9021

This laboratory analyzed only for TOC. They reported a value for the unspiked reagent water sample (D7). They were flagged High when their value for D7 was not subtracted from D8 and D9, but their performance improved to Acceptable when the correction was made (Table 6). This suggests laboratory contamination of the samples and greater care should be taken in sample handling. They had Acceptable performance for the effluent samples.

Laboratory 9023

Laboratory 9023 reported a value for DOC in the unspiked reagent water sample (D7). They had Acceptable performance for the spiked reagent water samples using the reported values for D8 and D9. Their rank shifted from Acceptable to Low with an Intercept problem when their reported result for D7 was subtracted from D8 and D9. This suggests contamination of sample D7.

They reported a low result for TOC in the unspiked reagent water sample (E7). Subtracting or not subtracting this value had no effect on the performance for TOC which was Acceptable in both matrices (Table 6).

Laboratory 9024

Laboratory 9024 was flagged Low with a Slope error in the reagent water samples for DOC. They did achieve Acceptable performance for the effluent samples. It is recommended that they confirm their calibration standards with reference materials.

Their results for TOC were Acceptable versus the target but were flagged as Warning Low versus the median for the reagent water samples. The value for S_w is smaller versus the median, resulting in a tighter area of acceptable performance. As this lab is only in the warning area, performance is considered acceptable. They had Acceptable performance for the effluent samples.

Laboratory 9025

Laboratory 9025 did not detect DOC in the low spike (D8) and had a very low result for D9. They had Acceptable performance for DOC in the effluent samples. However, this flag is based on the relationship of the spiked versus unspiked effluent samples. Their actual results for the effluent samples are lower than all of the other participants (Table 1 and Figure 9). This suggests a bias in their calibration standards and should be checked using reference materials.

They were assigned Acceptable performance for TOC in the reagent water samples versus the target when the reported results for D7 had not been subtracted from the D8 and D9 results (for some other participants). For all other combinations of evaluating the reagent water samples, this laboratory was flagged as Warning, Out of Control (Table 6). This is due to the low result of D9 relative to the interlaboratory mean and median and the target. They had Acceptable performance for the effluent samples.

Laboratory 9026

Laboratory 9026 did not detect DOC in the low spike (D8) and had a low result for sample D9. They achieved Acceptable performance for the effluent samples. As their results for the uncorrected effluent samples had good agreement with the other participants (Table 1 and not labelled in Figure 9), it appears that this laboratory's method is not sufficiently sensitive at the lower levels of the unspiked reagent water samples.

For TOC, they also were insufficiently sensitive at the lower levels to achieve good results for the reagent water samples (Table 2). They also did not distinguish between the unspiked effluent (E7) and the low effluent spike (E8). They were flagged as Low and/or Erratic for this test (Table 6). Further method development may improve the sensitivity of their method.

Laboratory 9027

Laboratory 9027 was flagged as Out of Control for DOC in the reagent water samples (Table 5). This is due to the high result they had for D8 relative to the interlaboratory mean and median and the target. They had Acceptable performance for the effluent samples.

They had Acceptable performance for TOC in the reagent water samples but were flagged Erratic for the effluent samples (Table 6). The flag for the effluent samples is due to their not distinguishing between the unspiked effluent (E7) and the low effluent spike (E8). This suggests that their method is not as sensitive for the matrix used in this interlaboratory study.

Laboratory 9028

Laboratory 9028 was flagged High with an Intercept problem for DOC in the reagent water samples. They should check their analytical system to determine how well they are setting their baseline. They had a warning of slight imprecision for the effluent samples when evaluated versus the target but were flagged as Out of Control versus the median. This is due to their result for E9 being higher than the interlaboratory median.

For TOC, Laboratory 9028 was flagged Warning High with Intercept problems versus the target in reagent water. They had Acceptable performance versus the median when the reported results for D7 had not been subtracted from the D8 and D9 results (for other participants). They were flagged High with Intercept problems when the correction for D7 was made (Table 6). As with DOC, they should check their analytical system to determine how well they are setting their baseline. They had Acceptable performance for TOC in the effluent samples.

Laboratory 9030

Laboratory 9030 reported a result for the unspiked reagent water sample (D7) for DOC. When evaluated versus the target, they had Acceptable performance. When evaluated against the median, they are ranked Warning High (Table 5). When their result for D7 was subtracted from D8 and D9 and evaluated versus the median, their rank improves to Acceptable. This apparent improvement in performance versus the median is affected by the lower interlaboratory median value for D9 relative to the target value. As the flag was a warning, overall performance is acceptable. They had Acceptable performance in the effluent samples.

They reported a low result for TOC in the unspiked reagent water sample (E7). Subtracting or not subtracting this value had no effect on the performance for TOC which was Acceptable in both matrices (Table 6).

Laboratory 9031

Laboratory 9031 required unpreserved samples for DOC and therefore was unable to analyze the study samples for this test. Their results for TOC were flagged High with an Intercept problem in the reagent water samples. They should check their analytical system to determine how well they are setting their baseline. They had Acceptable performance for the effluent samples.

Laboratory 9033

Laboratory 9033 reported results only for DOC. They added the following note regarding the lack of results for TOC: "According to MISA Regulations, TOC is required only if the TSS is greater than 15 mg/L".

They reported a value for the unspiked reagent water sample (D7). Their performance for DOC was very inconsistent. They were flagged Erratic versus the target and Acceptable with slight imprecision versus the median when the result for D7 was not subtracted from D8 and D9. When the result for D7 was subtracted, they were flagged Low with a Slope problem in both cases. For the effluent samples they were flagged Out of Control versus the median but were Acceptable with slight imprecision versus the target. More consistent application of their method should help improve their performance.

Laboratory 9034

This laboratory had Acceptable performance for both DOC and TOC in all samples in both matrices with no contamination in the unspiked reagent water sample (D7).

Laboratory 9038

This laboratory had Acceptable performance for both DOC and TOC in all samples in both matrices with no contamination in the unspiked reagent water sample (D7).

Laboratory 9039

Laboratory 9039 was flagged Warning Low for DOC in reagent water when the reported value for D7 (unspiked reagent water sample) was not subtracted from the results for D8 and D9 (other participants). They were assigned Acceptable performance when the correction for D7 was made (Table 5). When the correction is made, there is a change in the area of acceptable performance (Table 3), allowing for the change in Laboratory 9039's ranking. As the flags are in the warning area, their performance is considered acceptable. They had Acceptable performance for TOC in both matrices.

Laboratory 9040

Laboratory 9040 reported a value for the unspiked reagent water sample (D7). This value was higher than the result reported for the low spike (D8), so it was not subtracted from the spiked sample results for the evaluation procedure. As they had Acceptable performance for DOC in the reagent samples, it appears that sample D7 was contaminated. They were flagged as Warning Low for the effluent samples. Their method may be less sensitive for the matrix used in this study. They had Acceptable performance for TOC in both matrices.

Laboratory 9042

This laboratory had Acceptable performance for both DOC and TOC in all samples in both matrices with no contamination in the unspiked reagent water sample (D7).

Laboratory 9045

This laboratory analyzed only for TOC. The low spike in the reagent water sample (D8) was below their detection limit, so they were not included in the evaluation. Their result for D9 had good agreement with the target and was higher than the interlaboratory mean and median. They had Acceptable performance for the effluent samples.

Laboratory 9047

This laboratory had Acceptable performance for both DOC and TOC in all samples in both matrices with no contamination in the unspiked reagent water sample (D7).

Laboratory 9048

Laboratory 9048 had Acceptable performance for DOC in the reagent water samples. For the effluent samples, they had Acceptable performance versus the target but were flagged as Warning High versus the median. Their result for E9 was high compared to the interlaboratory median. They had Acceptable performance for TOC in both matrices.

5 REFERENCES

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6 APPENDIX 1 - TABLES AND FIGURES

Table 1	Dissolved Organic Carbon Results in mg/L
Table 2	Total Organic Carbon Results in mg/L
Table 3	Statistical Summary - Dissolved Organic Carbon
Table 4	Statistical Summary - Total Organic Carbon
Table 5	Performance Evaluation - Dissolved Organic Carbon
Table 6	Performance Evaluation - Total Organic Carbon
Figures 1 - 4	Youden Plots - Dissolved Organic Carbon
Figures 5 - 8	Youden Plots - Total Organic Carbon
Figures 9 - 10	Youden Plots - Effluent Samples Not Corrected for Background Levels

TABLE 1: DISSOLVED ORGANIC CARBON RESULTS IN mg/L

LAB CODE	D7	D8	D9	E7	E8	E9
SPIKE LEVEL	-	2	6	-	2	6
9002	N/A	N/A	N/A	N/A	N/A	N/A
9003	0.1<W	1.8	5.7	13	14.9	18.5
9005	<0.5 <DL	2.0	5.9	12.4	14.3	17.7
9006	<MDL	2.0	6.0	13.5	15.5	19.6
9010	<0.5	2.3	4.9	11.9	15.2	17.9
9011	0.35	2.40	6.28	14.39	16.36	20.00
9012	<0.5	3.1	5.7	15.5	16.0	24.7
9015	UNABLE TO REPORT RESULTS					
9016	<0.1	2.1	6.0	13.8	15.8	19.0
9019	<0.4	1.7	5.5	13.2	14.8	17.6
9020	0.21	2.34	5.79	13.87	15.38	18.7
9021	N/A	N/A	N/A	N/A	N/A	N/A
9023	0.82	2.2	5.7	18	20	24
9024	<0.5	1.6	4.9	13.6	16.2	19.4
9025	<1	<1	2	7	11	14
9026	<0.5	<0.5	4.1	15	17	21
9027	<0.5	3	6	16	18	21
9028	<0.5	2.8	6.7	16.2	17.3	22.3
9030	0.30	2.33	6.35	15.58	17.46	21.02
9031	*	*	*	*	*	*
9033	0.75	2.48	5.55	13.41	14.69	19.92
9034	<DL	2.1	6.0	15.8	17.3	20.9
9038	<0.1	2.0	5.8	15.0	17.1	20.7
9039	<0.2	1.8	5.4	12.2	13.7	17.6
9040	4.8	2.3	6.0	15	16	20
9042	<0.5	2.0	5.5	13.6	15.0	18.6
9045	N/A	N/A	N/A	N/A	N/A	N/A
9047	<0.4	2.2	6.1	12.1	13.7	17.6
9048	<0.50	2.09	5.75	13.5	15.8	20.2

* Laboratory 9031 required unpreserved samples to analyzed for DOC and was therefore unable to perform this analyses in this interlaboratory study.

TABLE 2: TOTAL ORGANIC CARBON RESULTS IN mg/L

LAB CODE	D7	D8	D9	E7	E8	E9
SPIKE LEVEL	-	2	6	-	2	6
9002	1<W	2<T	6	12	15	18
9003	N/A	N/A	N/A	N/A	N/A	N/A
9005	<0.5 <DL	2.0	5.8	12.8	14.8	18.6
9006	<MDL	1.97	6.0	13.7	15.9	19.7
9010	<0.5	2.5	4.9	13.4	19.7	29.2
9011	<0.5	2.45	6.45	15.79	17.92	21.61
9012	<1.0	4.3	7.3	19.3	20.3	25.6
9015	UNABLE TO REPORT RESULTS					
9016	N/A	N/A	N/A	N/A	N/A	N/A
9019	<0.4	2.3	5.7	15.4	15.8	17.2
9020	0.27	2.36	5.76	13.9	15.68	18.8
9021	3	5	10	21	22	27
9023	0.90	2.6	6.2	18	21	24
9024	<0.5	1.5	5.1	15.8	16.8	21.2
9025	<1	2	4	14	16	19
9026	<0.5	<0.5	4.2	19	19	22
9027	<0.5	2	6	15	15	20
9028	<0.5	2.8	6.7	16.2	18.3	22.3
9030	0.32	2.33	6.37	15.69	17.53	21.21
9031	<1.70	3.31	6.91	14.7	17.0	20.6
9033	**	**	**	**	**	**
9034	<DL	2.1	5.9	14.6	16.1	19.6
9038	<0.1	2.0	5.9	15.6	17.5	21.7
9039	<0.2	1.8	5.4	12.2	13.7	17.6
9040	5	2.3	6.1	15	17	21
9042	<0.5	2.0	5.5	14.1	15.0	19.8
9045	<5.0	<5.0	7.0	16.5	18.0	22.5
9047	<0.4	2.4	6.2	13.4	14.6	19.7
9048	<0.50	2.38	5.89	14.8	16.7	20.2

** This laboratory appended the following note: "According to MISA Regulations, TOC is required only if TSS is greater than 15 mg/L." They did not report results for this test for this interlaboratory study.

TABLE 3 - STATISTICAL SUMMARY - DISSOLVED ORGANIC CARBON

	REAGENT WATER				EFFLUENT	
SAMPLE	D8	D9	D8-D7	D9-D7	E8	E9
TARGET	2	6	2	6	2	6
MEAN	2.026	5.567	1.925	5.466	1.873	5.766
MEDIAN	2.15	5.77	2.035	5.725	1.9	5.55
STD DEV	0.709	0.907	0.700	0.907	0.701	0.936
n	22	24	22	24	24	24
SELECTED MEDIAN	2.1	5.8	2.04	5.9	1.9	5.5
SELECTED STD DEV	0.278	0.255	0.063	0.215	0.385	0.616
n	20	19	11	17	21	23
S_H/S_L =	0.916		3.389		1.598	
S_w - TARGET	0.185		5.161 %		0.419	
S_w - MEDIAN	0.159		4.989 %		0.310	

TABLE 4 - STATISTICAL SUMMARY - TOTAL ORGANIC CARBON

	REAGENT WATER				EFFLUENT	
SAMPLE	D8	D9	D8-D7	D9-D7	E8	E9
TARGET	2	6	2	6	2	6
MEAN	2.258	6.049	2.078	5.870	1.777	5.849
MEDIAN	2.3	5.95	2.01	5.9	1.9	5.8
STD DEV	0.997	1.103	0.822	0.789	1.194	2.263
n	23	25	23	25	23	25
SELECTED MEDIAN	2.3	6	2	5.9	1.9	5.85
SELECTED STD DEV	0.260	0.572	0.290	0.789	0.296	0.429
n	19	22	21	25	15	22
S_H/S_L =	2.201		2.726		1.448	
S_w - TARGET	12.96 %		10.04 %		0.515	
S_w - MEDIAN	8.75 %		8.46 %		0.475	

TABLE 5 - PERFORMANCE EVALUATION - DISSOLVED ORGANIC CARBON

	REAGENT WATER				EFFLUENT	
	TARGET		MEDIAN		TARGET	MEDIAN
CODE	D8 VS D9	D8-D7 VS D9-D7	D8 VS D9	D8-D7 VS D9-D7	E8-E7 VS E9-E7	
9003	A	A	A	A	A	A
9005	A	A	A	A	A	A
9006	A	A	A	A	A	A
9010	OC	ER	OC	ER	WOC	OC
9011	A	A	WHS	A	A	A
9012	OC	OC	OC	OC	ER	ER
9016	A	A	A	A	A	A
9019	WLS	WLI	WLI	WLI	WLS	WLS
9020	AI	A	A	A	A	A
9023	A	Li	A	LI	A	A
9024	LS	LS	Ls	Ls	A	A
9025	Le	Le	Le	Le	Hi	Hi
9026	Le	Le	Le	Le	A	A
9027	OC	OC	OC	OC	A	A
9028	HI	HI	HI	HI	AI	OC
9030	A	A	WHS	A	A	A
9033	ER	LS	AI	WLS	AI	OC
9034	A	A	A	A	A	A
9038	A	A	A	A	A	A
9039	WLS	A	WLI	A	A	A
9040	A	AI	A	A	WLI	WLI
9042	A	A	A	A	A	A
9047	A	A	A	A	A	A
9048	A	A	A	A	A	WHS

KEY TO SUMMARY TABLE

A	Acceptable Performance
WLI	Warning: Biased low, Probable intercept problem
WLS	Warning: Biased low, Probable slope problem
WOC	Warning: Out of control - one result erratic
LS	Biased low, Probable slope problem
Ls	Biased low, Possible slope problem
Le	Biased low and/or erratic
HS	Biased high, Probable slope problem
Hs	Biased high, Possible slope problem
He	Biased high and/or erratic
ER	Both results erratic
AI	Warning: Slight imprecision
WHI	Warning: Biased high, Probable intercept problem
WHS	Warning: Biased high, Probable slope problem
LI	Biased Low, Probable intercept problem
Li	Biased low, Possible intercept problem
L	Biased low
HI	Biased high, Probable intercept problem
Hi	Biased high, Possible intercept problem
H	Biased high
OC	Out of control - one result erratic

TABLE 6 - PERFORMANCE EVALUATION - TOTAL ORGANIC CARBON

	REAGENT WATER				EFFLUENT	
	TARGET		MEDIAN		TARGET	MEDIAN
CODE	D8 VS D9	D8-D7 VS D9-D7	D8 VS D9	D8-D7 VS D9-D7	E8-E7 VS E9-E7	E8-E7 VS E9-E7
9002	A	A	A	A	A	A
9005	A	A	A	A	A	A
9006	A	A	A	A	A	A
9010	Al	Al	Al	ER	Hs	H
9011	A	A	A	WHI	A	A
9012	OC	Hi	Hi	Hi	A	Al
9019	A	A	A	A	LS	LS
9020	A	A	A	A	A	A
9021	Hi	A	H	Al	A	A
9023	A	A	A	A	A	A
9024	A	A	WLI	WLI	A	A
9025	A	WOC	WOC	WOC	A	A
9026	Le	Le	Le	Le	Le	Le
9027	A	A	A	A	ER	ER
9028	WHI	WHI	A	HI	A	A
9030	A	A	A	A	A	A
9031	HI	HI	HI	HI	A	A
9034	A	A	A	A	A	A
9038	A	A	A	A	A	A
9039	A	A	A	A	A	A
9040	A	A	A	A	A	A
9042	A	A	A	A	A	A
9045	N/A	N/A	N/A	N/A	A	A
9047	A	A	A	A	A	A
9048	A	A	A	Al	A	A

FIGURE 1: DISSOLVED ORGANIC CARBON

LOW SAMPLE VS HIGH SAMPLE IN REAGENT WATER

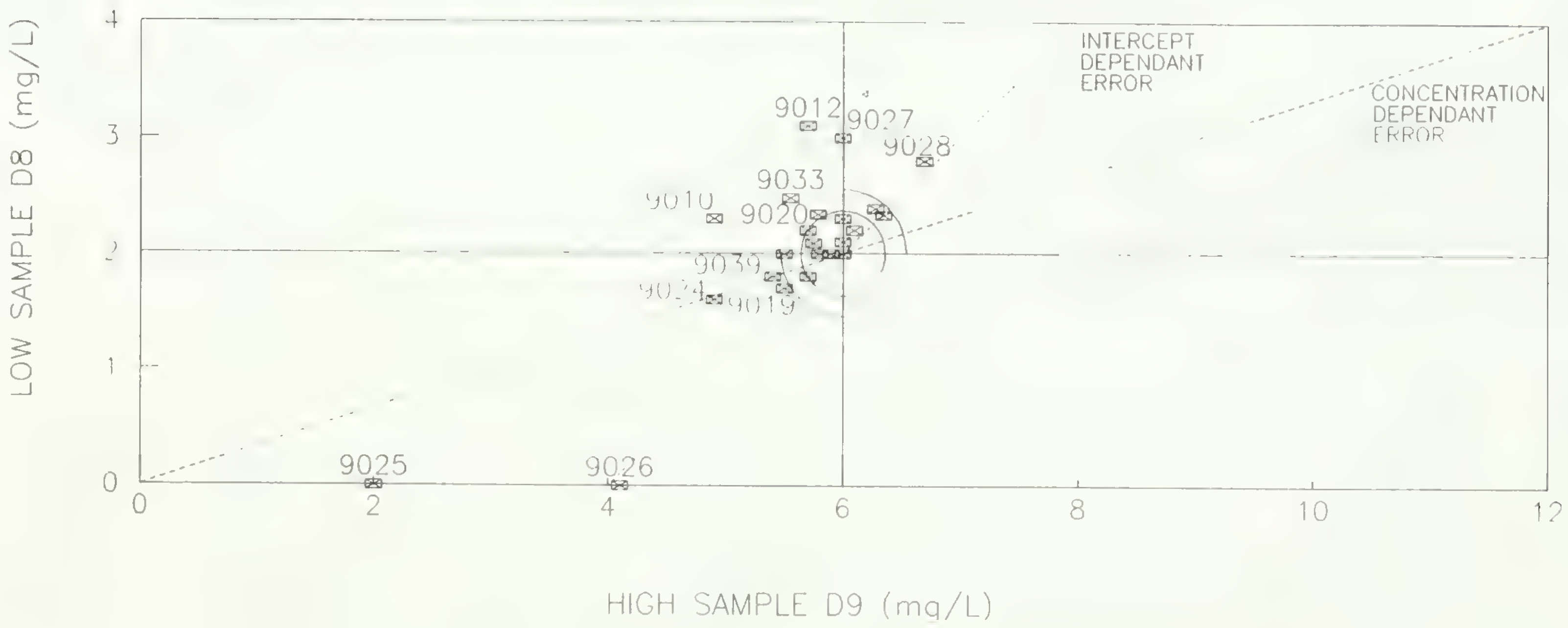
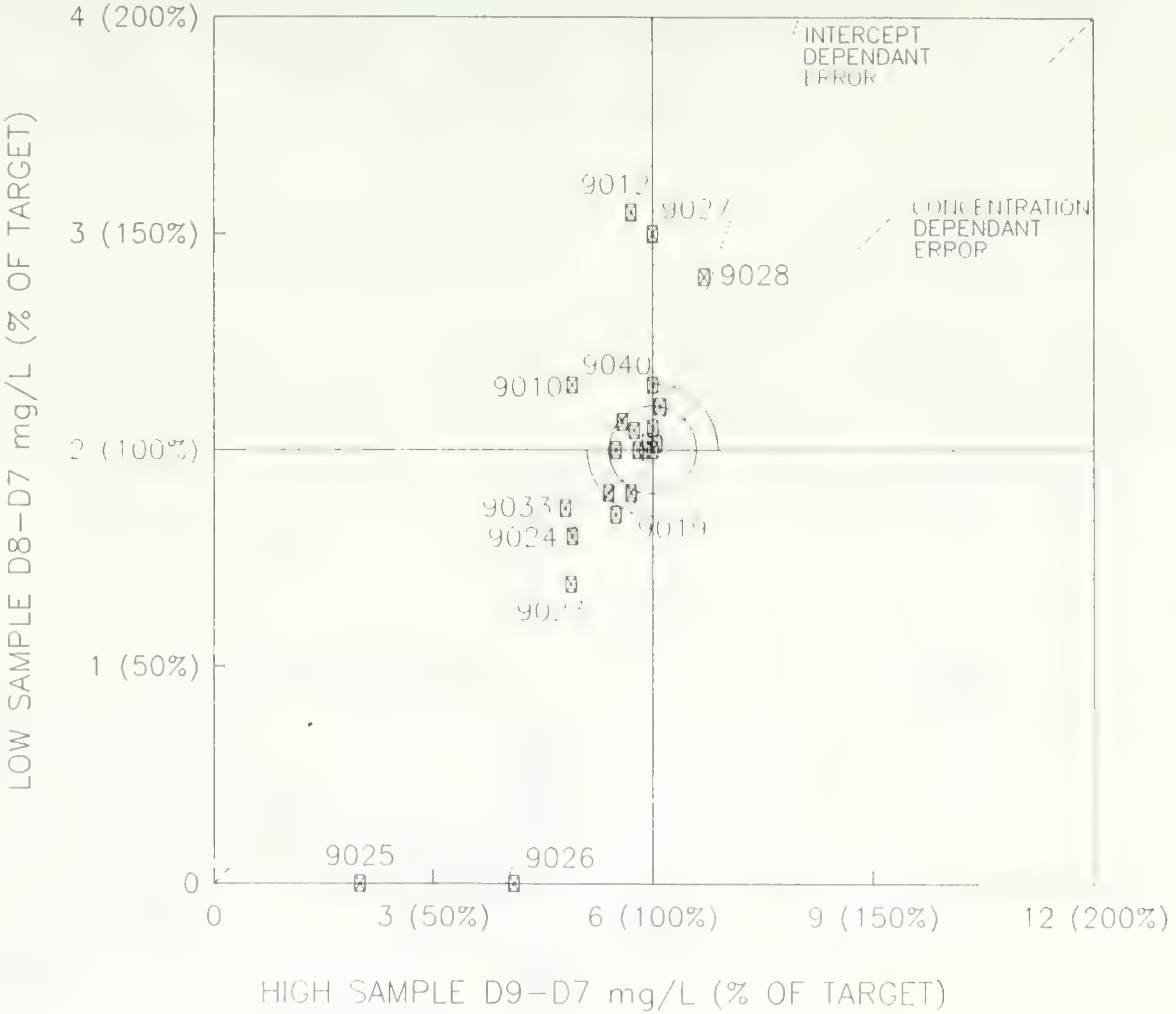


FIGURE 1A: DISSOLVED ORGANIC CARBON

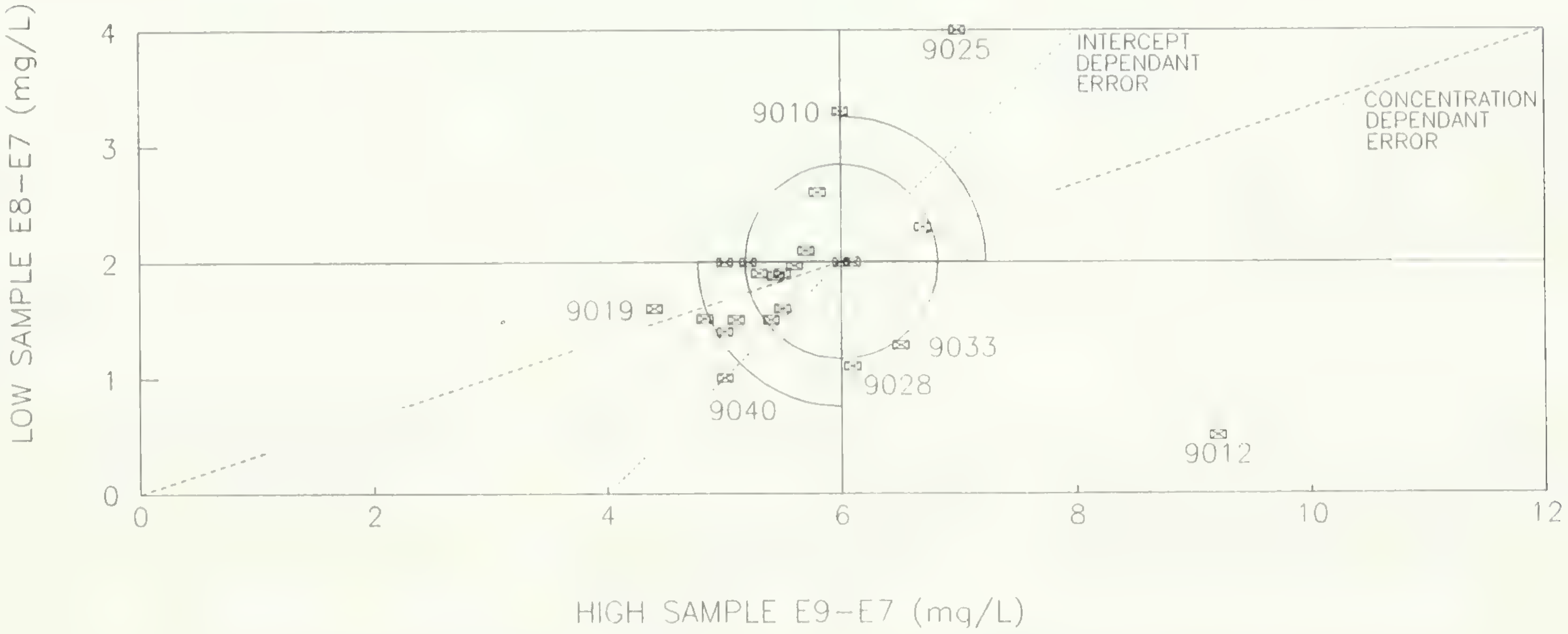
LOW SAMPLE VS HIGH SAMPLE IN REAGENT WATER



RESULTS CORRECTED FOR REPORTED VALUE IN UNSPIKED SAMPLE (D7).

FIGURE 2: DISSOLVED ORGANIC CARBON

LOW SAMPLE VS HIGH SAMPLE IN EFFLUENT



RESULTS CORRECTED FOR REPORTED VALUE IN UNSPIKED SAMPLE (E7).

FIGURE 3: DISSOLVED ORGANIC CARBON

LOW SAMPLE VS HIGH SAMPLE IN REAGENT WATER

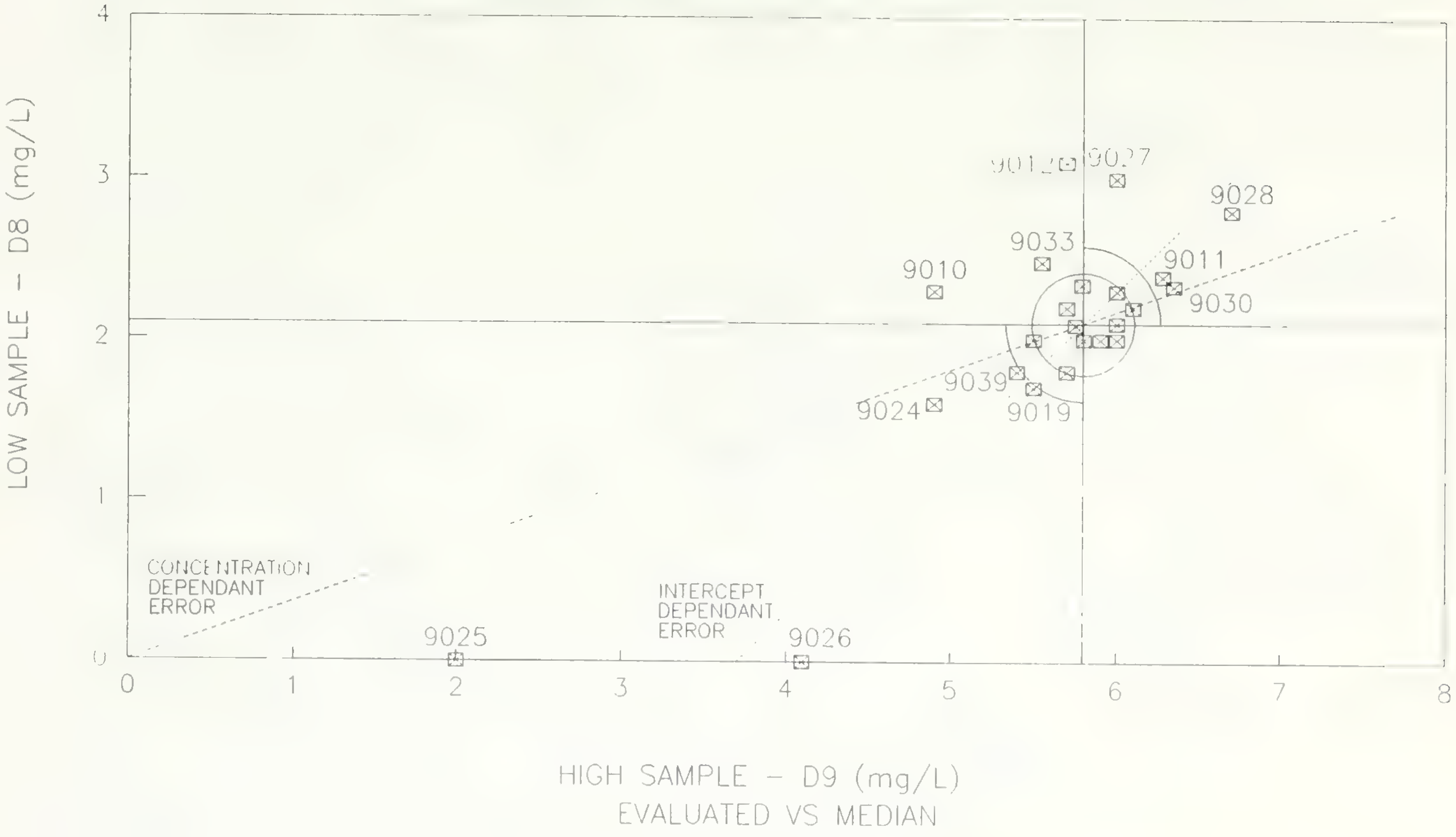
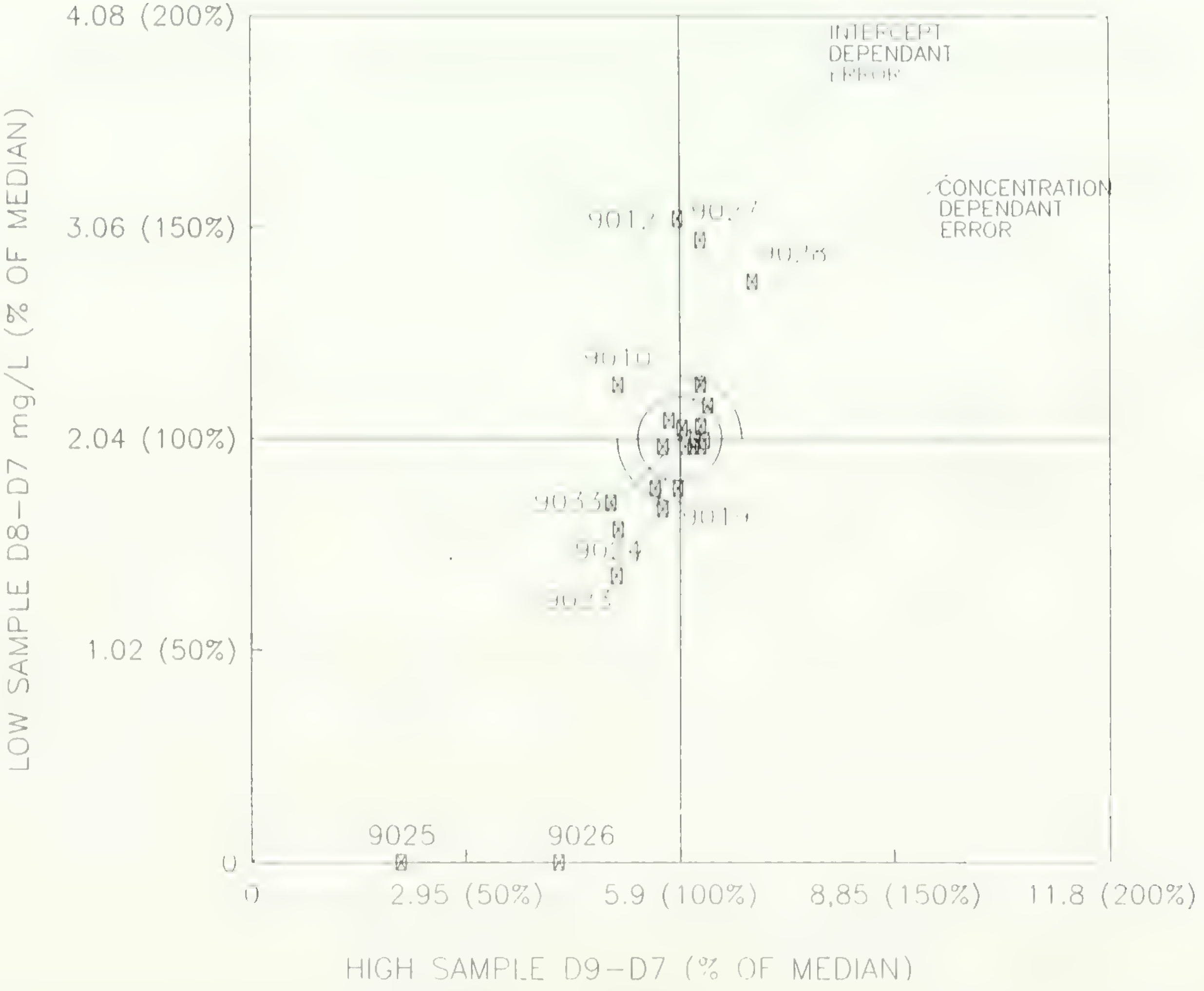


FIGURE 3A: DISSOLVED ORGANIC CARBON

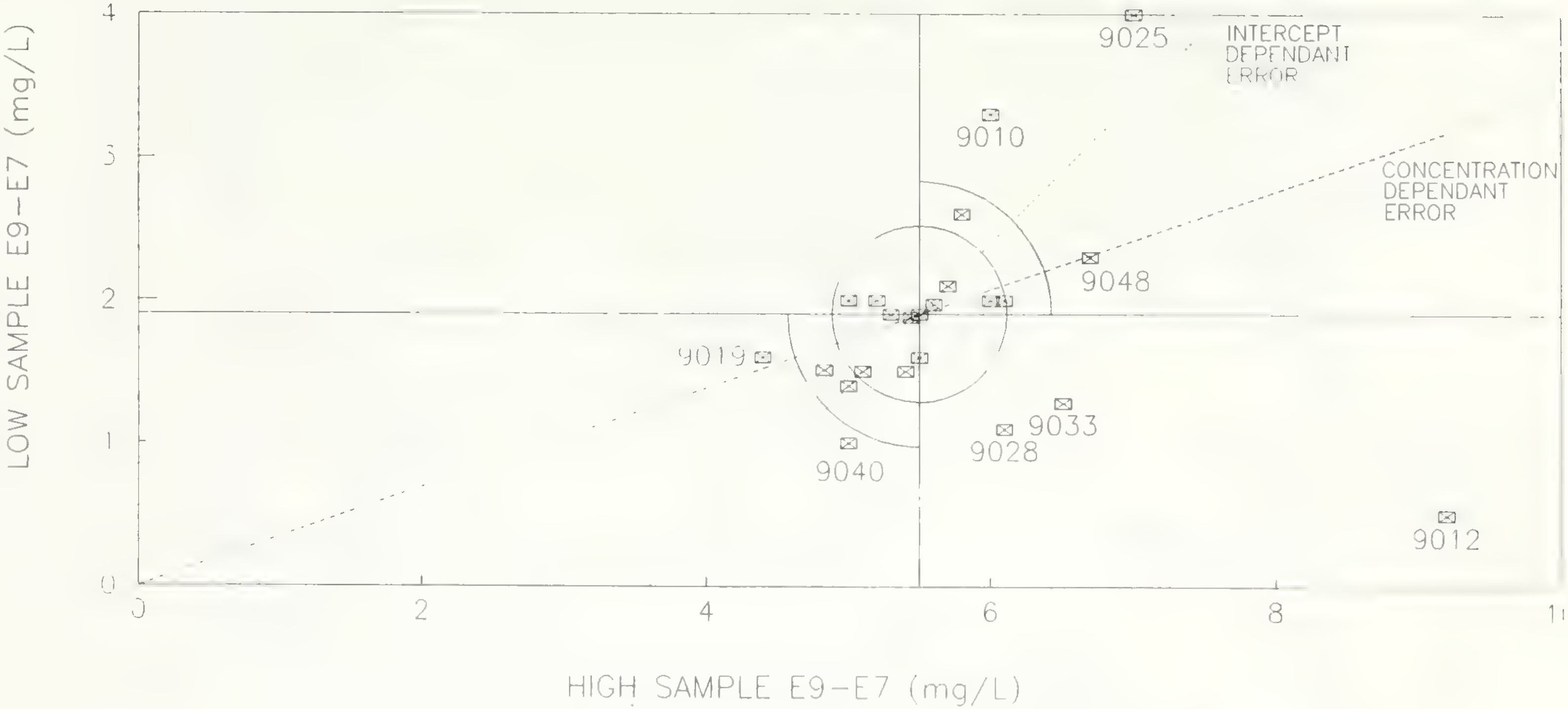
LOW SAMPLE VS HIGH SAMPLE IN REAGENT WATER



RESULTS CORRECTED FOR REPORTED VALUE IN UNSPIKED SAMPLE (D7).

FIGURE 4: DISSOLVED ORGANIC CARBON

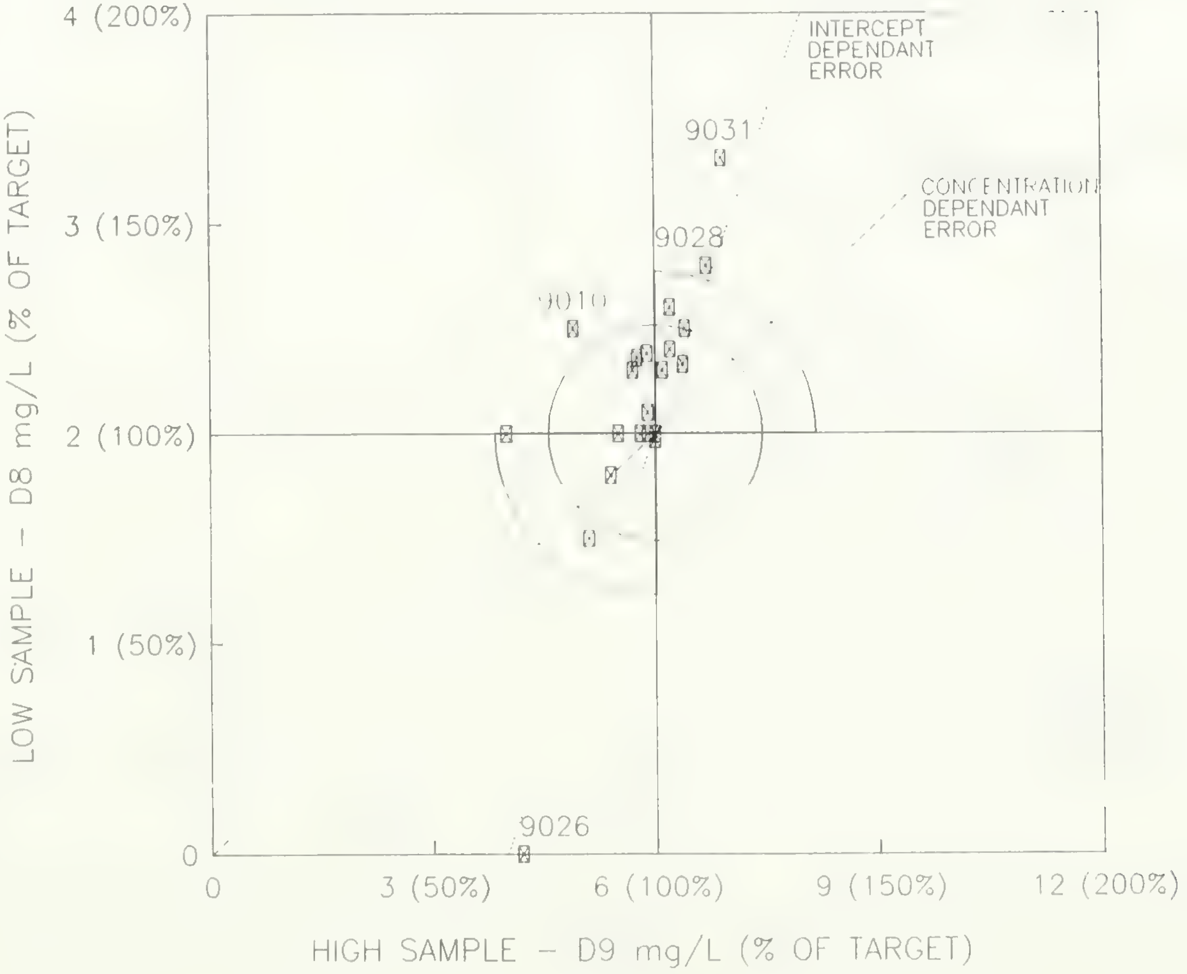
LOW SAMPLE VS HIGH SAMPLE IN EFFLUENT



RESULTS CORRECTED FOR REPORTED VALUE IN UNSPIKED SAMPLE (E7).

FIGURE 5: TOTAL ORGANIC CARBON

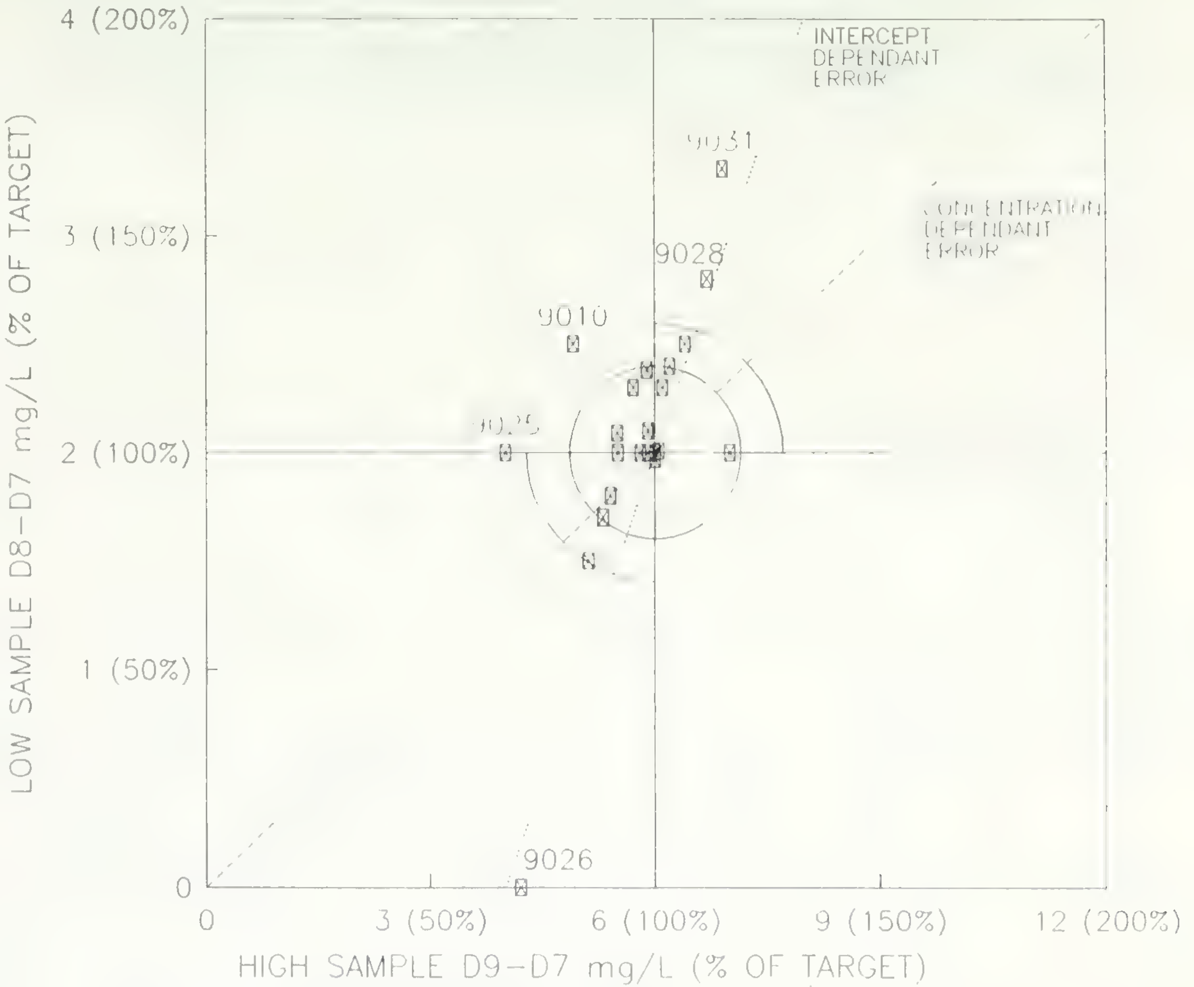
LOW SAMPLE VS HIGH SAMPLE IN REAGENT WATER



LABORATORY 9012 IS OUTSIDE THE SCALE OF THE DIAGRAM

FIGURE 5A: TOTAL ORGANIC CARBON

LOW SAMPLE VS HIGH SAMPLE IN REAGENT WATER

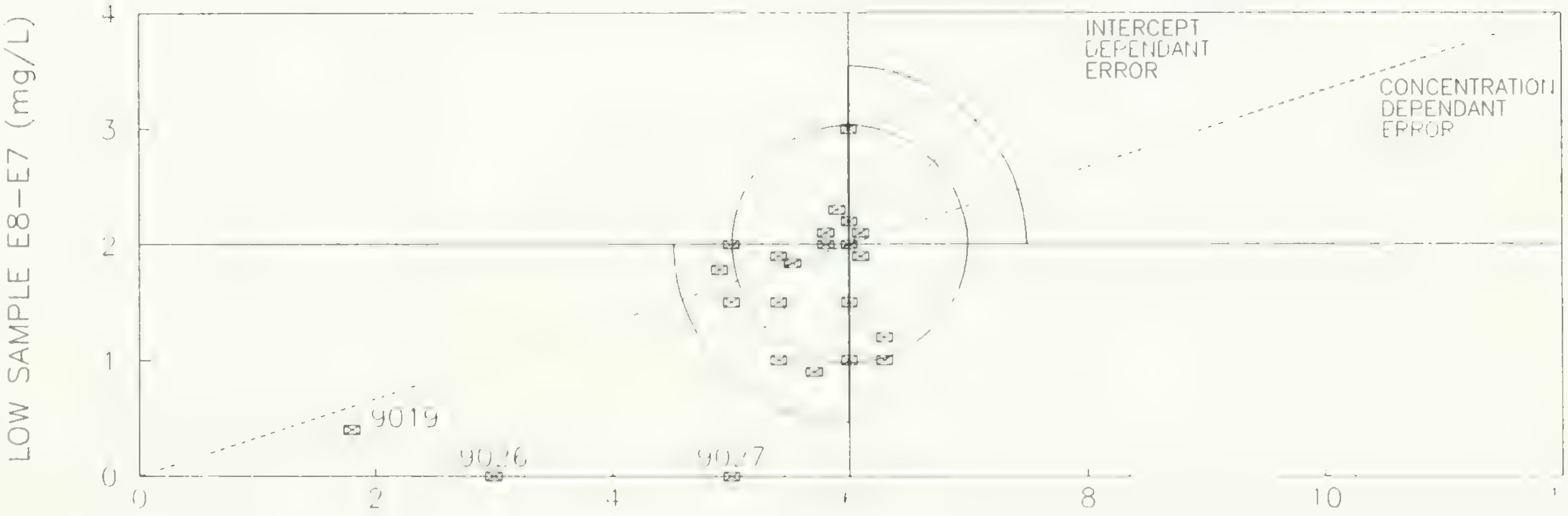


RESULTS CORRECTED FOR REPORTED VALUE IN UNSPIKED SAMPLE (D7).

LABORATORY 9012 IS OUTSIDE THE SCALE OF THE DIAGRAM

FIGURE 6: TOTAL ORGANIC CARBON

LOW SAMPLE VS HIGH SAMPLE IN EFFLUENT



HIGH SAMPLE E9–E7 (mg/L)

RESULTS CORRECTED FOR REPORTED VALUE IN UNSPIKED SAMPLE (E7).

FIGURE 7: TOTAL ORGANIC CARBON

LOW SAMPLE VS HIGH SAMPLE IN REAGENT WATER

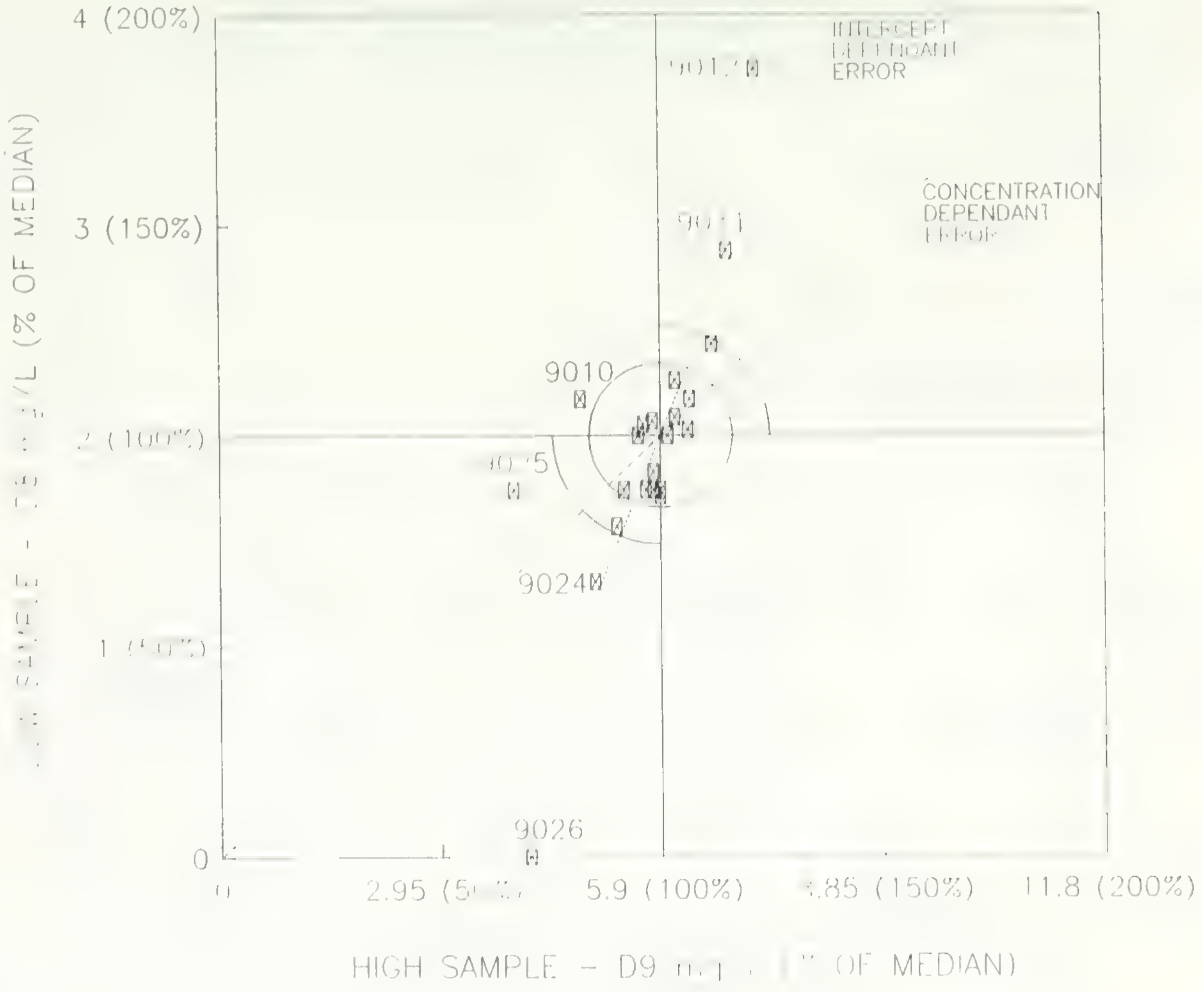
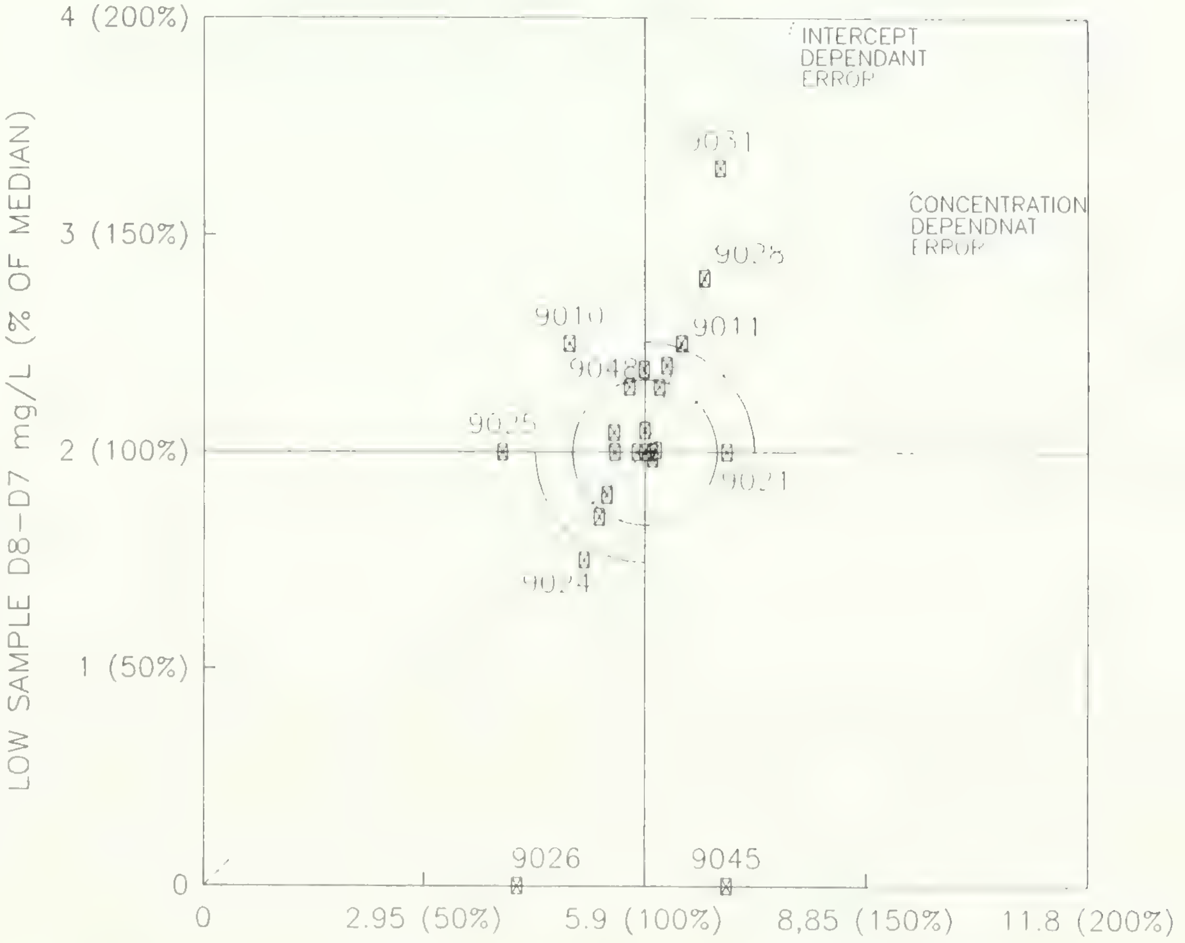


FIGURE 7A: TOTAL ORGANIC CARBON

LOW SAMPLE VS HIGH SAMPLE IN REAGENT WATER



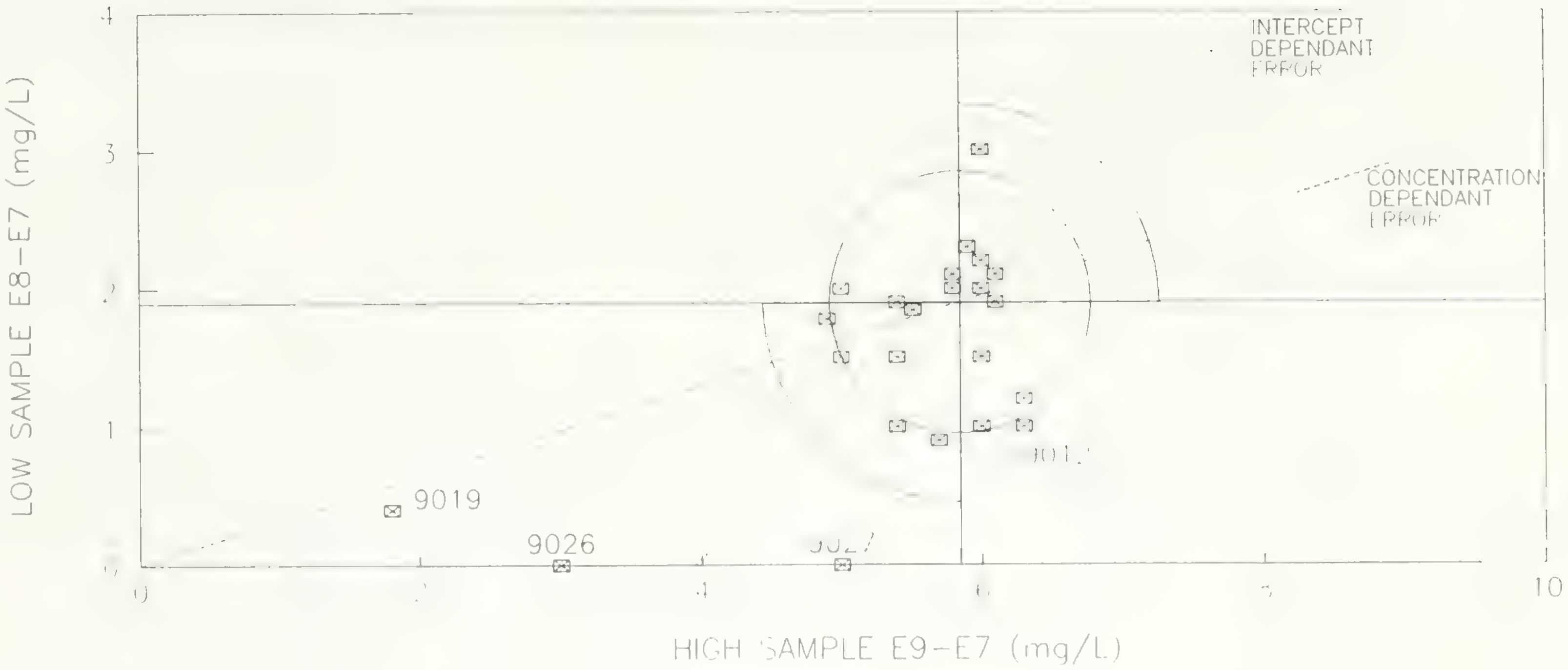
HIGH SAMPLE D9–D7 mg/L (% OF MEDIAN)

RESULTS CORRECTED FOR REPORTED VALUE IN UNSPIKED SAMPLE (D7).

FIGURE 8: TOTAL ORGANIC CARBON

LOW SAMPLE VS HIGH SAMPLE IN EFFLUENT

EVALUATED VS MEDIAN



RESULTS CORRECTED FOR REPORTED VALUE IN UNSPIKED SAMPLE (E7).

FIGURE 9: DISSOLVED ORGANIC CARBON

SPIKED EFFLUENT SAMPLES

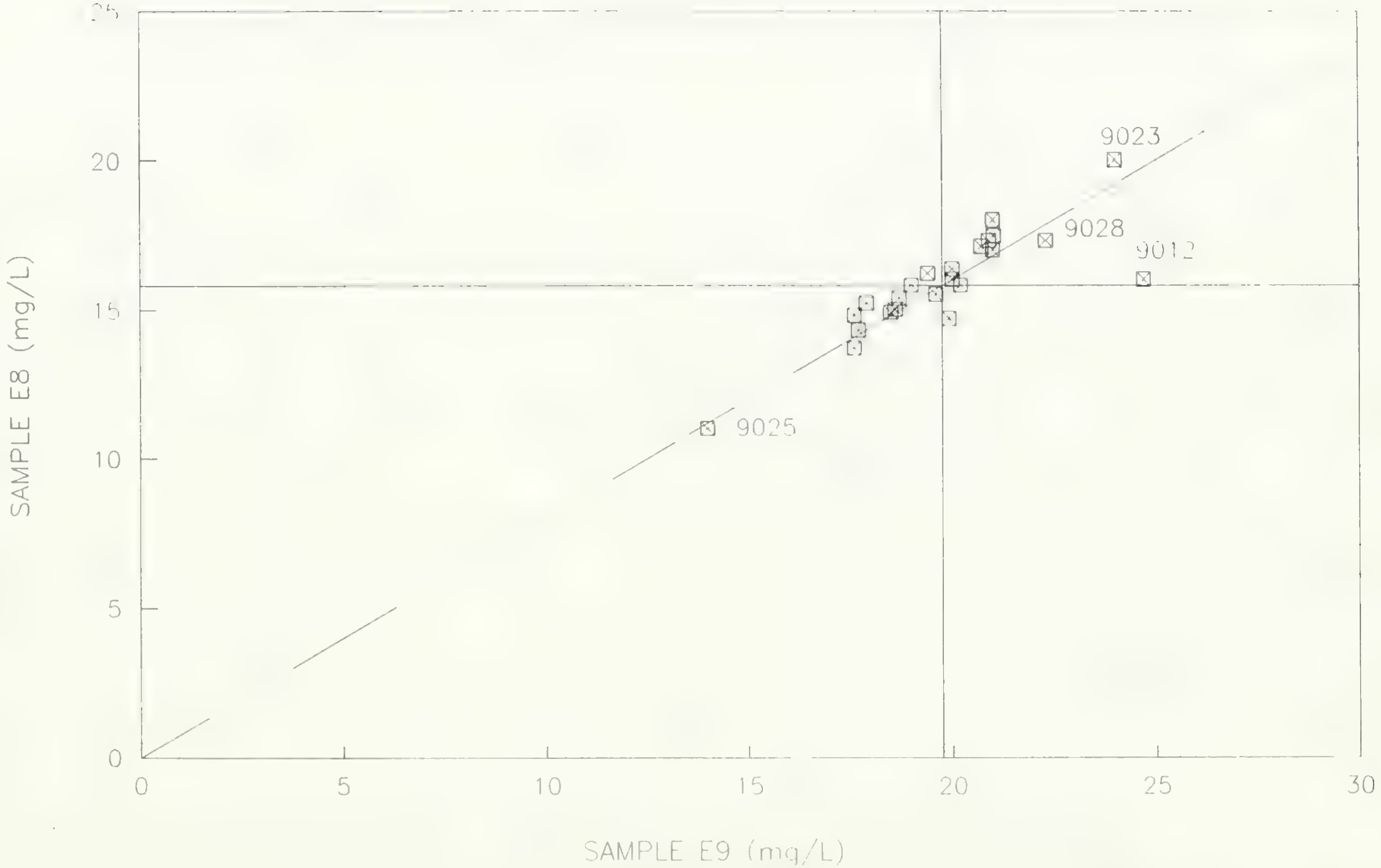
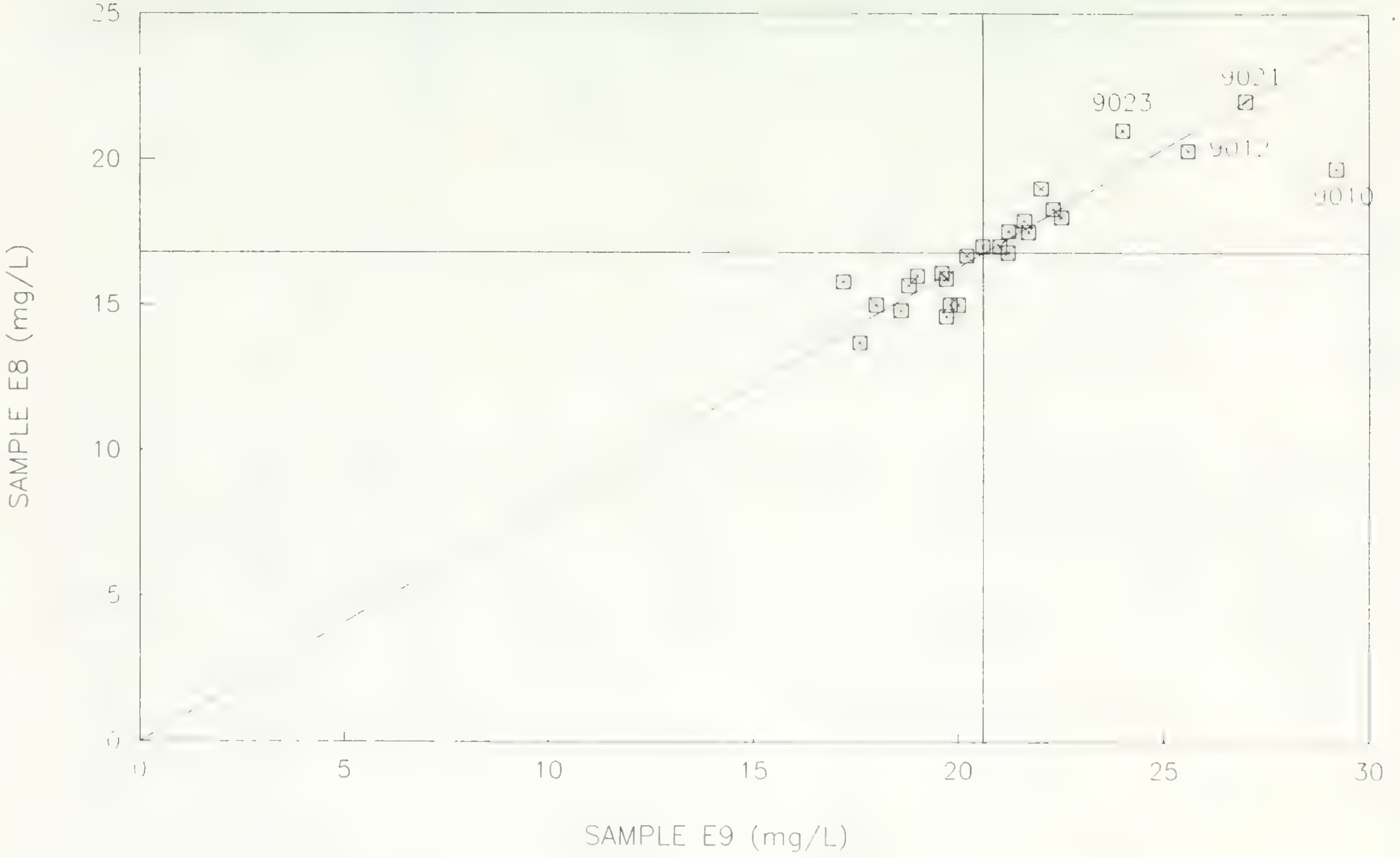


FIGURE 10: TOTAL ORGANIC CARBON

SPIKED EFFLUENT SAMPLES



7 APPENDIX 2 - LIST OF PARTICIPANTS AND CORRESPONDENCE

LIST OF PARTICIPANTS

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Manitoba Environment
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Contact: Larry Prokopanko/Donna Collins
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Regional Municipality of Waterloo
Engineering Laboratory
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Cambridge, Ontario
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Contact: David Andrews
(519) 885-9500

MOE INTERLABORATORY VARIABILITY STUDY NOTIFICATION FOR THE ANALYSIS OF TRACE INORGANIC COMPOUNDS

STUDY NO. 91-3, STUDY 91-4, & STUDY 91-5

INTRODUCTION

Laboratories receiving this notification are invited by Environment Ontario, Laboratory Services Branch, to participate in three different interlaboratory variability studies of spiked reagent water and spiked mixed effluent, conducted using MISA analysis protocols. Laboratories interested in participating in this program, scheduled for three successive weeks commencing with the week of October 21, 1991, should contact Sylvia Cussion at (416) 235-5842 of Environment Ontario to confirm participation. Written confirmation (attached response sheet) must be submitted to Environment Ontario no later than October 15, 1991 (FAX: (416) 235-6110).

BACKGROUND

This study is being conducted to assist laboratories in assessing their analytical performance in conjunction with the MISA program. All procedures should follow those principles and protocols outlined in the MISA regulations (Ontario Reg. 695/88 as amended to Ontario Reg. 533/89). These studies target three different MISA test groups. Separate sets of samples will be distributed for each analytical test, one set each per week, for three successive weeks. Sample sets will include spiked reagent water samples and effluent matrix samples. Laboratories may choose to participate in only one, two, or all three of the studies.

The sample schedule is as follows:

TUDY	TEST	MISA Group	No. of Samples	Matrix	Preservative	Study Date
1-3	Phenolics	14	3	Reagent Water	H_2SO_4	October 22, 1991
	(4AAP)		3	Effluent	H_2SO_4	October 22, 1991
1-4	Solvent	25	3	Reagent Water	None	October 29, 1991
	Extractables		2	Effluent	None	October 29, 1991
1-5	Organic Carbon	5a & b	3	Reagent Water	H_2SO_4	November 4, 1991
	DOC & TOC		3	Effluent	H_2SO_4	November 4, 1991

NOTE: If any of the above preservatives present an analytical problem, please indicate this on the response form.

Participating laboratories are expected to analyze the samples within the time limits specified in Schedule 2 of the general MISA regulations (Ontario Reg. 695/88). Blank reporting forms will be provided with the samples. Results for all analyses are to be reported within thirty (30) days of receipt of the samples to Sylvia Cussion at the following address:

Environment Ontario
Laboratory Services Branch
Quality Management Unit
125 Resources Rd., P.O. Box 213
Rexdale, Ontario
M9W 5L1

SUMMARY OF RESULTS

All participating laboratories will be assigned a unique identification code. All laboratories reporting results will receive a complete set of the results where they will be identified only by the identification code. Recommendations made by Environment Ontario will be included in the final report. Results will remain confidential and will only be released with the written permission of the individual participants.

It is the intent of this interlaboratory study (along with others) to assess the interlaboratory variability and detection capability for a broad range of organics and inorganics.

MOE INTERLABORATORY VARIABILITY STUDY NOTIFICATION FOR THE ANALYSIS OF TOTAL PHENOLS, OIL & GREASE, AND DOC & TOC

STUDY 91-3, STUDY 91-4 & STUDY 91-5

We will participate for the following study(ies):

	YES	NO	Minimum Sample Volume Required
Study 91-3 Phenolics (4AAP)	☐	☐	____________
Study 91-4 Solvent Extractables	☐	☐	____________
Study 91-5 Organic Carbon (DOC & TOC)	☐	☐	____________

For the completeness of our records, and to avoid any shipping delays, please fill in the following:

Mailing Address:	Shipping Address:
______________________	______________________
______________________	______________________
______________________	______________________
______________________	______________________

Contact Person: ____

Telephone: ____

Please return this response form by October 15, 1991 to:

Sylvia Cussion
Environment Ontario
Laboratory Services Branch
Quality Management Unit
125 Resources Rd., P.O. Box 213
Rexdale, Ontario, M9W 5L1

FAX: (416) 235-6110

Quality Management Unit
(416) 235-5842
FAX: (416) 235-6110

November 5, 1991

TO: PARTICIPANTS OF INTERLABORATORY STUDY 91-5

Please find enclosed six 500 mL plastic bottles for the analysis of Dissolved Organic Carbon (DOC) and Total Organic Carbon (TOC), MISA Test Groups 5a and 5b. The samples are preserved with H_2SO_4 and are to be used for both tests. The samples are labelled as follows:

Distilled water:	D7	D8	D9
Effluent:	E7	E8	E9

If you are missing any of the above items, please contact me at the above phone number immediately.

As stated in the notification distributed September 27, 1991, samples should be analyzed using the principles and protocols outlined in the MISA General Regulation (Ontario Reg. 695/88, as amended to Ont. Reg. 533/89). Store all samples in a refrigerator at 4 degrees Celcius until ready for analysis. Time limit for storage is 10 days.

To ensure timely release of a summary report, results are to be submitted by December 13, 1991. Report forms to be used are included with the samples. Please identify all sample results with your lab identification number and the sample numbers described above. Please contact me if there are any problems or questions re the interlaboratory study. Thank you for your participation.

Your lab identification number is:

Sincerely,

Sylvia Cussion
Lab Quality Audit Scientist

INTERLABORATORY STUDY 91-5

DISSOLVED AND TOTAL ORGANIC CARBON

REPORT FORM

NOVEMBER 5, 1991

REPORT DUE DATE: DECEMBER 13, 1991

LAB IDENTIFICATION CODE:

SAMPLE	DOC (mg/L)	TOC (mg/L)
D7		
D8		
D9		
E7		
E8		
E9		

SAMPLE PREPARATION PRINCIPLES:

INSTRUMENTAL MEASUREMENT METHOD PRINCIPLES:

Quality Management Unit
(416) 235-5842

January 15, 1992

TO: PARTICIPANTS OF INTERLABORATORY STUDY 91-5

Thank you for your participation in Interlaboratory Study 91-5 for the analysis of Dissolved Organic Carbon and Total Organic Carbon. Please find attached a copy of the table of results. Please note that some participants analyzed the samples for one test only. If there are any transcription errors, please contact me no later than February 7, 1992. A report evaluating the results will be prepared and a copy provided to all participants.

Your lab identification number is:

Sincerely,

Sylvia Cussion
Lab Quality Audit Scientist
(416) 235-5842
FAX (416) 235-6110

ATTACHMENT